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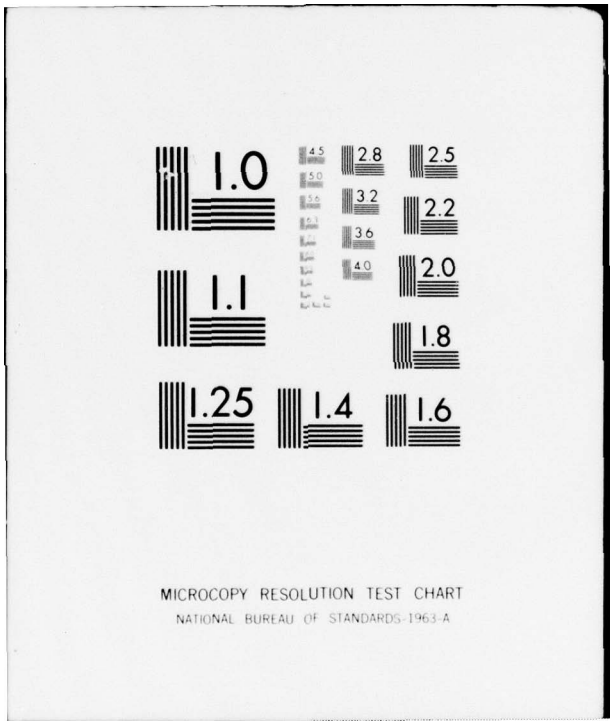
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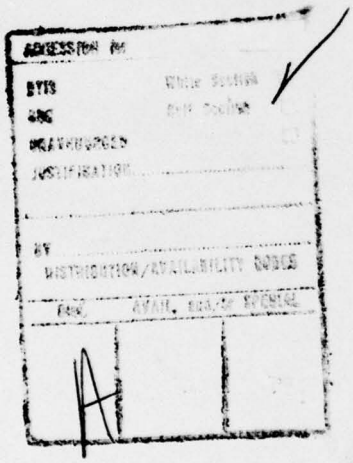
JUDITH L. HOUSE and GARY L. HAGNAUER
POLYMER AND CHEMISTRY DIVISION

December 1976

ARMY MATERIALS AND MECHANICS RESEARCH CENTER
Watertown, Massachusetts 02172



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ABSTRACT

The liquid exclusion mechanism for separation in liquid chromatography is evaluated for fingerprinting and monitoring the compositions of hydraulic fluids. A variety of hydraulic fluids are examined and testing procedures are developed. The utility of liquid chromatography in trace analysis and in separating specific fluid components for characterization or identification is demonstrated.

I. INTRODUCTION

Most hydraulic fluids are complex mixtures consisting of the petroleum or non-petroleum base formulated with various additives which may be present in trace amounts or constitute up to 20% by weight of the fluid. Fluids with the same specifications may have very different compositions if they are obtained from different sources or if the supplier changes formulations. The composition of fluids often change during use, by contamination with other fluids, or by aging or separation during storage. Such differences or changes in the chemical composition of a hydraulic fluid are known to have catastrophic effects on the overall performance of hydraulic systems, compatibility of the fluid with system components, and longevity of the fluid.

There are currently no satisfactory inspection procedures for evaluating the chemical composition of hydraulic fluids. Existing inspection procedures for fluids are cursory and very qualitative. Applications exist not only for fluid monitoring but also for fingerprinting and perhaps evaluating the chemical composition of hydraulic fluids. Although many techniques (wet chemical, infrared, NMR, GC, TLC, SEM, X-ray) have been considered, they are generally found to be either lacking in versatility, speed, accuracy, reproducibility or are too costly, large, and complex for monitoring fluids.

High performance liquid chromatography (HPLC) is perhaps the most versatile technique that might be used for analyzing hydraulic fluids. A variety of separation mechanisms (liquid exclusion, liquid-liquid, liquid-solid, and ion-exchange) and detectors (RI, UV, VIS, IR) are available. The primary requirement is that the sample be soluble in some carrier solvent. HPLC is a fast, quantitative, analytical technique that requires small amounts of sample and is ideally suited for fluid monitoring and trace analysis. Recent advances in liquid chromatography technology have resulted in improved pumping systems, better column packings, new detectors, and miniaturization of components. Furthermore, the basic instrumentation is relatively low in cost ($3500 to $6000) and simple to operate.

In this report the liquid exclusion mechanism for separation in HPLC is evaluated for fingerprinting and monitoring the compositions of hydraulic fluids. Different types of hydraulic fluids are examined and testing procedures are developed. Modifications in instrument design are made to improve analysis and reliability. Additionally, the utility of HPLC in trace analysis and in separating specific fluid components for characterization or identification is demonstrated.

II. EXPERIMENTAL

Liquid exclusion chromatography is used for the compositional analysis of the hydraulic fluids (HPLC) and for characterizing the high molecular weight (polymer) component(s) of the fluids (GPC). The hydraulic fluids examined

are listed in Table I. Since the fluids are completely miscible with tetrahydrofuran (THF), freshly distilled THF is used as the carrier solvent for both the HPLC and GPC studies.

The basic design of the HPLC system is depicted in Figure 1. An LDC model 709 pumping station* consisting of a reciprocating pump with pulse damper and pressure gage controls the solvent flow rate (2.8 ml/min and 450 psi at room temperature). A flow restrictor is placed in the line between the pump and sample injector to provide a more constant flow rate. The hydraulic fluid sample is injected into a manual valve and loop (0.2 ml) assembly using a 1-ml B-D tuberculin syringe and Swinny adapter with 5μ Teflon

Table 1. HYDRAULIC FLUIDS

Fluid Specification	Condition/Source
FA PD-5136	New - Royal Lubricants
FA PD-5136	New - Bray Oil
FA PD-5136	Used - Bray Oil, after 221 miles and 32 hours operation
MIL-H-6083C	New - Penn Refining Co.
MIL-H-6083C	Used - Penn Refining Co., after 2195 miles and 466 hr operation
MIL-H-5606C	New - Royal Lubricants
MIL-H-83282	New - Mobil Oil
	New - Skydrol 500B, phosphate ester
APG PD#1	New - Emery Industries
APG PD#1	New - Conoco
MIL-L-10295A	New - Gulf Oil

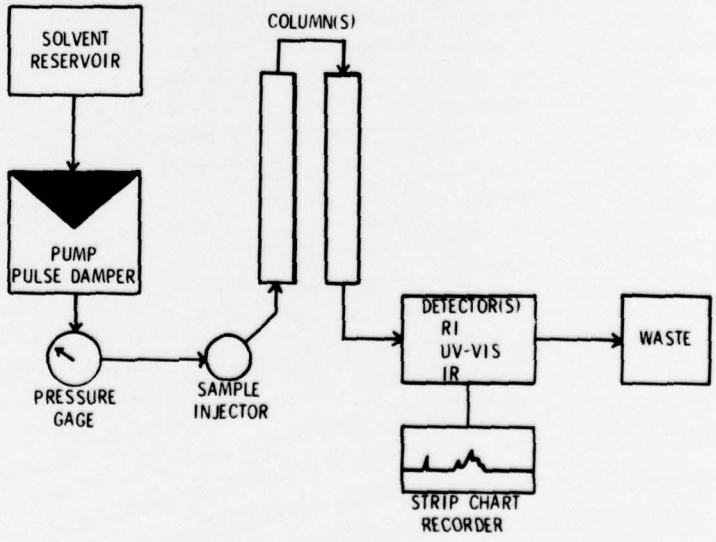

Figure 1. Basic design of HPLC.

*Laboratory Data Control, Riviera Beach, FL

Millipore filter. For the preparative work with this system a 0.4 ml sample loop is used. The column set consists of three 4-ft X 3/8-in. Styragel columns with porosity ratings of 100, 100, and 80-150 Å as designated by the manufacturer (Waters Assoc., Milford, MA). Under the above conditions this column set has an efficiency of 340 plates/ft using o-dichlorobenzene as the standard. The eluent from the columns is detected by two different monitors: UV Duomonitor model 1222 and RI Refractomonitor model 1103.* The UV monitor detects absorbances at 254 and 280 nm. Both monitors are differential detectors with flowing solvent references. An LDC model 3402 chromatographic strip chart recorder measures and records signals from the monitors using a chart speed of 4 mm/min. The mean time per sample analysis is about 45 minutes and chromatographic profiles are highly reproducible.

A Waters ANAPREP gel permeation chromatograph (GPC) is used to analyze the high molecular weight component(s) of the hydraulic fluids. When necessary, several high molecular weight fractions of a fluid are collected and concentrated to provide sufficient material for characterization. The column set consists of four 4-ft X 3/8-in. Styragel columns with porosity ratings of 10^3, 10^4, 10^5, and 10^6 Å and has a plate count of 1200 plates/ft. The solvent flow rate is 1 ml/min at 25 C. Narrow distribution polystyrene standards are used to calibrate the columns and standard techniques are used to calculate sample molecular weights based on the polystyrene calibration curve.

III. RESULTS AND DISCUSSION

Fingerprinting

Chromatograms obtained in analyzing different types of petroleum base fluids are illustrated in Figure 2. These chromatograms partially fingerprint the chemical compositions of the various fluids. Although all components of a given fluid are not necessarily resolved or detected by the monitors, distinct differences in the chromatograms are observed. Aside from direct comparison by overlaying chromatograms, certain characteristics of the chromatograms aid in identifying and distinguishing between the compositions of hydraulic fluids. The number, molecular size, and relative quantity of detected fluid components are indicated by peak position, shape, and area. For example, in Figure 3 sample injection is indicated at zero time. As time t increases, fluid components are separated in the columns and are detected upon elution from the columns in order of decreasing molecular size, assuming no interaction with column substrate material. All components may not be distinguishable because of similarities in their molecular size, limitations in resolving power of the columns, or detector insensitivity. The highest molecular weight components (polymer) are detected 23.3 minutes (93 mm) after injection; whereas very small molecules are last to appear, 46.5 min (186 mm). The peak height or integrated peak area depends on the component's chemical structure and is directly proportional to its concentration.

*Laboratory Data Control, Riviera Beach, FL

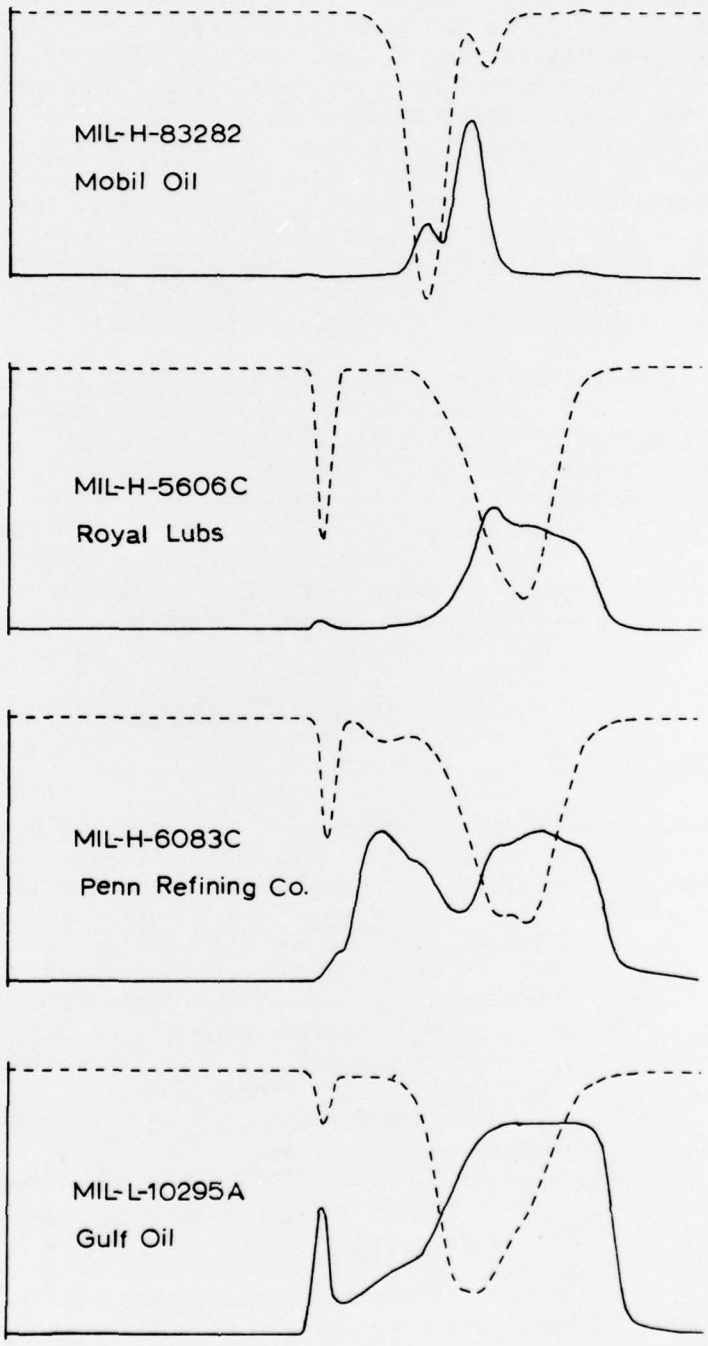

Figure 2. Chromatograms of petroleum base fluids using RI (----------) and 254 nm UV (———) monitors.

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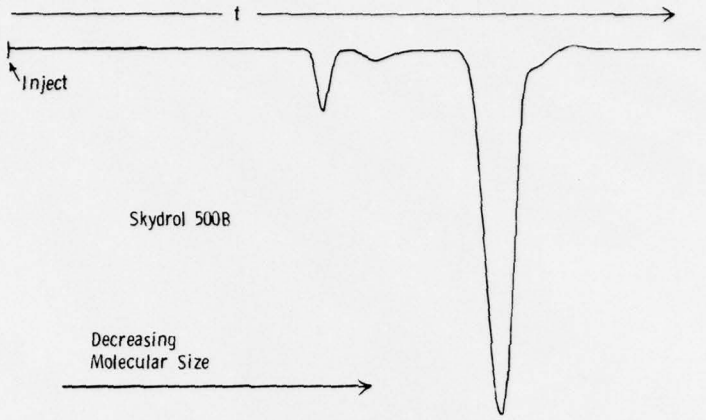

Figure 3. Chromatogram of Skydrol 500B phosphate ester.

The RI monitor measures the refractive index difference between THF and the separated sample component in the THF solution. No peak will be observed if the refractive indices of the separated component and solvent are very similar. The RI peak of a given component will be displaced in the positive or negative direction depending on its refractive index value relative to that of the solvent. The downward displacement of peaks shown in Figure 3 indicates that the components have a refractive index greater than that of THF (n_o = 1.404). The UV monitor measures the difference in relative absorbance between flowing streams of the solvent (reference) and separated components in solution. Generally the UV monitor will be more sensitive than the RI in detecting trace amounts of components. The relative absorbances at 254 nm and 280 nm may also be used in fingerprinting or classifying the separated components as shown in Figure 4. Certain components may absorb more at one wavelength than another, resulting in observable differences in relative peak heights. Otherwise undetected or masked components may thereby become apparent when monitored at different wavelengths.

Figures 4 and 5 illustrate how the "fingerprint" of a hydraulic fluid might be recorded and compared. The RI profiles of the FA PD-5136 fluids are quite similar; whereas the UV profiles readily distinguish compositional differences in the two fluids. Compositional differences are apparent in both the RI and the UV profiles of the APG-PD#1 fluids.

Monitoring

Figure 6 illustrates how HPLC may be used to monitor hydraulic fluids for contaminants or for changes in composition during use. The chromatograms indicate differences between compositions of the new fluids as obtained from the suppliers and the used fluids after being run in a hydraulic system. In interpreting the chromatograms, knowledge of maximum allowable compositional changes (contaminant levels, loss of volatiles, chemical changes due to degradation, oxidation, etc.) that a hydraulic fluid system will tolerate must be determined. Operating conditions for fluid separation and monitoring specific compositional changes in the fluid may then be established.

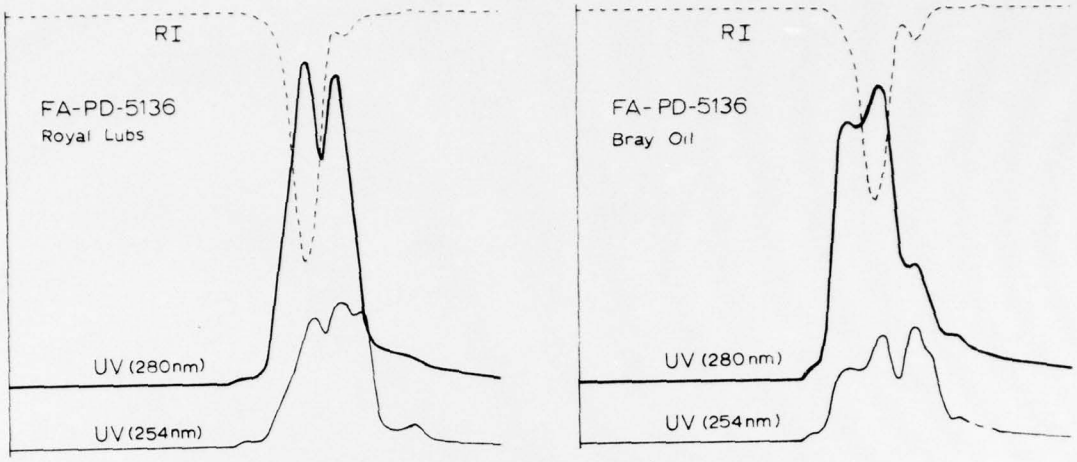

Figure 4. Compositional profiles of FA PD-5136 fluids.

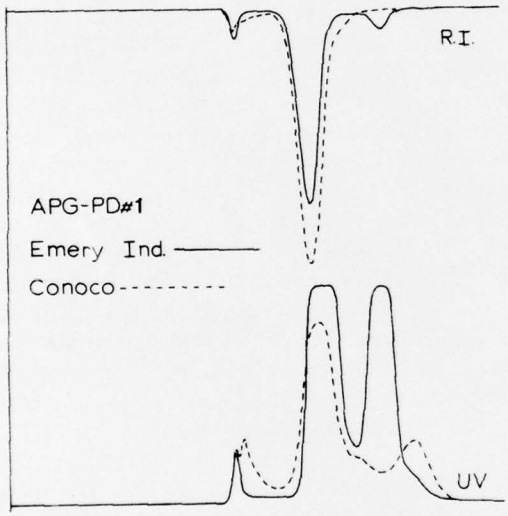

Figure 5. Compositional profiles of **APG-PD#1 fluids. (280 nm UV)**

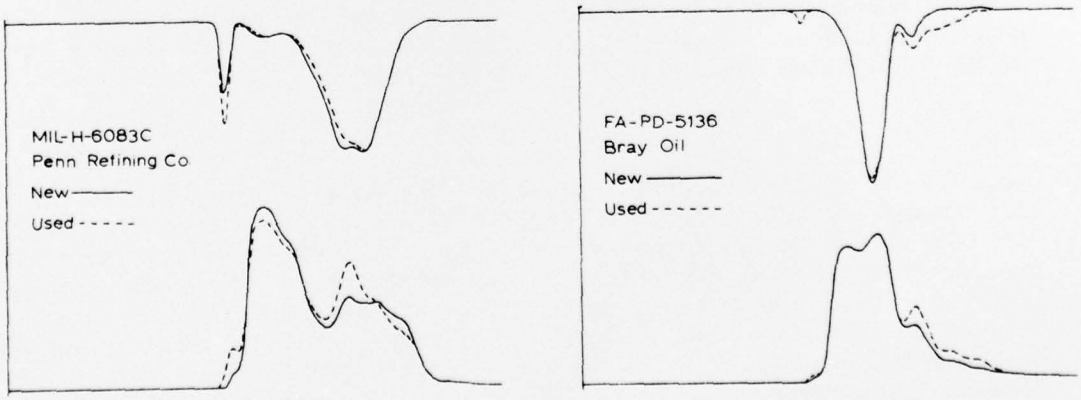

Figure 6. Fluid monitoring using PHLC (280 nm UV).

Specific Component Analysis

Hydraulic fluids frequently contain polymeric components as viscosity extenders. The polymer's concentration, as well as its molecular weight and molecular weight distribution, is important to the performance of the hydraulic fluid. HPLC is an effective technique for separating and quantitatively analyzing the polymeric component(s) in hydraulic fluids. As mentioned previously, the high molecular weight polymeric component(s) are first to elute from the columns and generally appear as a peak 23.3 minutes after sample injection. The height or integrated area of the polymer peak is proportional to the polymer's concentration. In addition, the polymer component may be separated for identification purposes or for molecular weight analysis by running the HPLC in the preparative mode; i.e., by using a larger loop size (0.4 ml) for sample injection and collecting the polymer as it leaves the monitor. In some cases, several injections and combining of polymer fractions may be required to provide sufficient sample for analysis. The polymer component may then be identified by spectroscopic techniques or its molecular weight may be evaluated by GPC. As an example of specific component analysis, the polymer component of Skydrol 500B is separated for molecular weight analysis (Figure 7). Since the identity of the polymer is unknown, the molecular weight analysis is based directly on the polystyrene calibration. The apparent number- and weight- average molecular weights are $\bar{M}_n^* = 43,100$ and $\bar{M}_w^* = 104,000$. For the Royal Lubs MIL-H-5606C sample, the apparent molecular weights are $\bar{M}_n^* = 34,900$ and $\bar{M}_w^* = 76,000$. The compositional profile (Figure 5) of the Conoco APG-PD#1 fluid suggests two high molecular weight components. Indeed the two components are separable by GPC consisting of a high molecular weight polymer, $\bar{M}_n^* = 191,000$ and $\bar{M}_w^* = 549,000$, and a relatively low molecular weight species, $\bar{M}_n^* = 4,270$ and $\bar{M}_w^* = 5,290$.

The compositional profiles of the new and used Penn Refining Co. MIL-H-6083C fluid (Figure 6) indicate a change in the polymer peak height. Contamination with another hydraulic fluid, loss of volatile fluid components, or thermo-oxidative effects during use may produce such a change. Specific component analysis by GPC may be used to determine whether the molecular weight of the fluid's polymeric component is affected. In this case, GPC analysis indicates no significant change in the molecular weight and molecular weight distribution of the polymer. The apparent molecular weights are $\bar{M}_n^* = 28,800$ and $\bar{M}_w^* = 80,500$ for the new polymer and $\bar{M}_n^* = 29,600$ and $\bar{M}_w^* = 71,200$ for the used polymer.

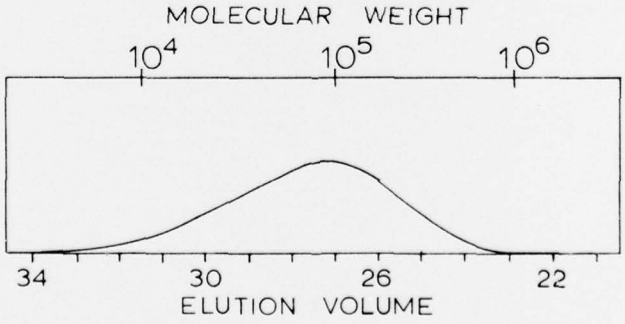

Figure 7. GPC chromatogram of polymer component in Skydrol 500B.

IV. FUTURE WORK

Liquid exclusion is only one of the separation mechanisms that might be used with HPLC for the compositional analysis of hydraulic fluids. Future work involves evaluating elution gradient HPLC and other "new" detectors and column substrate materials for "fingerprinting" hydraulic fluids. Particularly, infrared detectors should be evaluated for water and specific functional group analysis. Case studies will be made in fingerprinting and monitoring hydraulic fluids from actual, operative hydraulic systems to demonstrate the versatility of HPLC and to evaluate the precision of the technique. It is proposed that the instrument design and testing method accommodate two types of inspection packages: 1) for the rapid monitoring of fluid composition, specific fluid components or contaminants, and 2) for the detailed "fingerprinting" of newly procured or suspect fluids. It is also proposed that a versatile and mobile unit be designed to meet these specifications.

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